



INSTITUTE FOR ENGINEERING THERMODYNAMICS
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Laboratory course report
**Optical Diagnostics in Energy and Process Engineering -
Application of TDLAS**

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1 Introduction

A rather simple, but yet effective optical technique for analyzing and determining the species, concentration, pressure, temperatures or density of materials is the Tunable Laser Absorption Spectroscopy (TDLAS). This optical method relies on the specific and characteristic light absorption of substances. It used a manipulated laser to generate a bandwidth of wavelength. With that one can have a look at complete spectras and the absorption behavior over them. It can be grouped into the laser spectroscopy methods. Specific applications are particle technology (determining particle densities or concentrations), environmental technology, material composition or other technical fields.

This paper is documenting and discussing an experiment, more specific a laboratory course, with this measurement method. It is carried out at the Institute for engineering thermodynamics (LTT) of the FAU Erlangen-Nuremberg at its site in Erlangen-Tennenlohe.

The assignment, description of the equipment and procedure and further details about the Lab Course are described in the given handbook [1].

- Results

In the results section, the results are to be presented appropriately and discussed briefly in each case. Make sure that the dark signals of channel "a" and "b" and the measured calibration data are taken into account.

1. Calibration curve for the two measuring channels.

-> Presentation of the calibration curve in an xy-diagramm (x = wavelength; y = calibration factor) additional value table for the wavelength-dependent calibration factors. Discussion of further influences and suggestions for improvement.

2. Pure substance spectra and reference spectrum (see 4.1)

-> Presentation of the 2 absorption spectra of the pure substances in an xy-diagramm (x = wavelength; y = calibration factor) and labelling of the wavelengths for determination of the extinction coefficient..

3. Determination of the extinction coefficient of [EMIM][EtSO₄] and DMSO for positions 2, 5, 7 und 9 (siehe 4.2)
-> Tabulation of the results
 4. Discussion of the measurement uncertainty for [EMIM][EtSO₄] and suggestions for improvement.
 5. Discussion of errors.
- Conclusion

2 Theoretical basics

The following theoretical basics are summarized from the standard literature in optics [2]–[5] and more specifically Raman application [6], [7]. In addition the given handbook [1] is used as well.

The central principle TDLAS relies on, is the absorption of light. Next to absorption there are more processes which can occur with an incoming photon or light beam, such as scattering, reflection, or diffraction. In this case the focus is only on the first. Absorption of photons, or electromagnetic radiation, can only occur, if the struck molecule matches the energy of the photon with the energy difference of two of its discrete energy levels. This is called the resonance condition. This energy can be expressed as

$$E = h \cdot \nu.$$

The absorption can be visualized when hitting the molecule with a bandwidth light source. When analyzing the spectrum after the interaction, there are gaps or narrow dark bars occurring in the spectrum. Smaller molecules tend to form discrete lines, larger molecules more washed out reduction of the light spectrum. When using lower energy containing infrared radiation (IR), more vibrational or rotational states in the electronic ground state of the molecule are penetrated. In comparison the usage of higher energy containing ultra violet light, the electronic ground state can be elevated as well, and other unwanted effects such as fluorescence can occur. With the IR absorption, the generated spectrum and its gaps are acting like a fingerprint for the molecule. Species determination is easily possible that way.

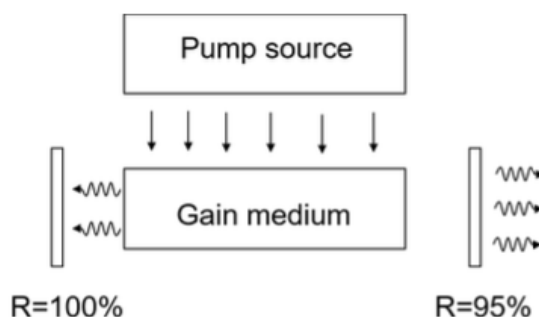


Figure 2.1: Simple working principle of a laser; simplified components [1]

To precisely analyse the absorption spectra, a bandwidth light source is not pleasant to use. A better way is the usage of a wavelength tunable laser. This can e.g. be achieved with a

Littrow laser arrangement (see Figure 2.2), like in this experiment. This arrangement allows a tunable output wavelength through a variable placement of a grating, thus realised through an easy to control electrical motor. The diffraction grating reflects the first diffraction order of the laser beam back and forms a standing wave. It is used with its easy positioning for creating the needed amplification (the basic laser principle is sketched in Figure 2.1). After reaching the laser threshold, the laser beam is coupled into a glass fibre and guided to the experimental setup. As a pump source a laser diode is utilized. The emitted basis wavelength is further tunable through the diode current and the temperature.

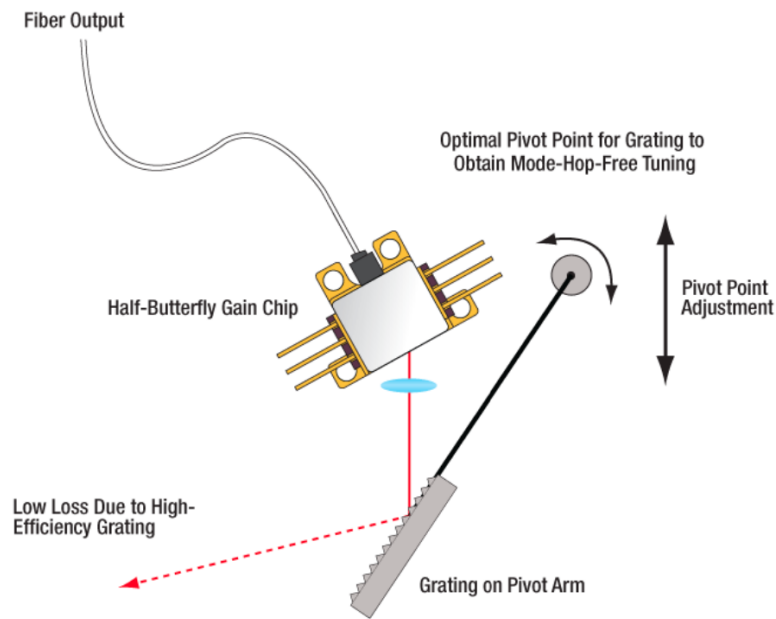


Figure 2.2: Tunable littrow laser arrangement [1]

3 Experimental setup

Following chapter shall describe the used experimental setup. The strategy of the data evaluation and result calculation shall be explained.

3.1 Measurement setup and preparations

A scheme of the measurement setup is illustrated in Figure 3.1. Controlling the whole setup, mainly the Littrow laser system and the chopper, and reading out the oscilloscope for data saving, is a computer. The laser signal is generated with a tunable diode laser setup (see Figure 2.2) for generating a defined bandwidth (between 1891 nm and 2022 nm) of wavelengths (see chapter 2 for a functional description). This signal is split into two beams and measurement paths. The chopper is acting as a pulser, so that the substance molecules and the photo diodes can relax and deexcite between the absorption processes. Between the chopper and the photodiode the probe is placed. After an intensification of the signal, it is read out with an oscilloscope. As a consequence the measurement is transmittive.

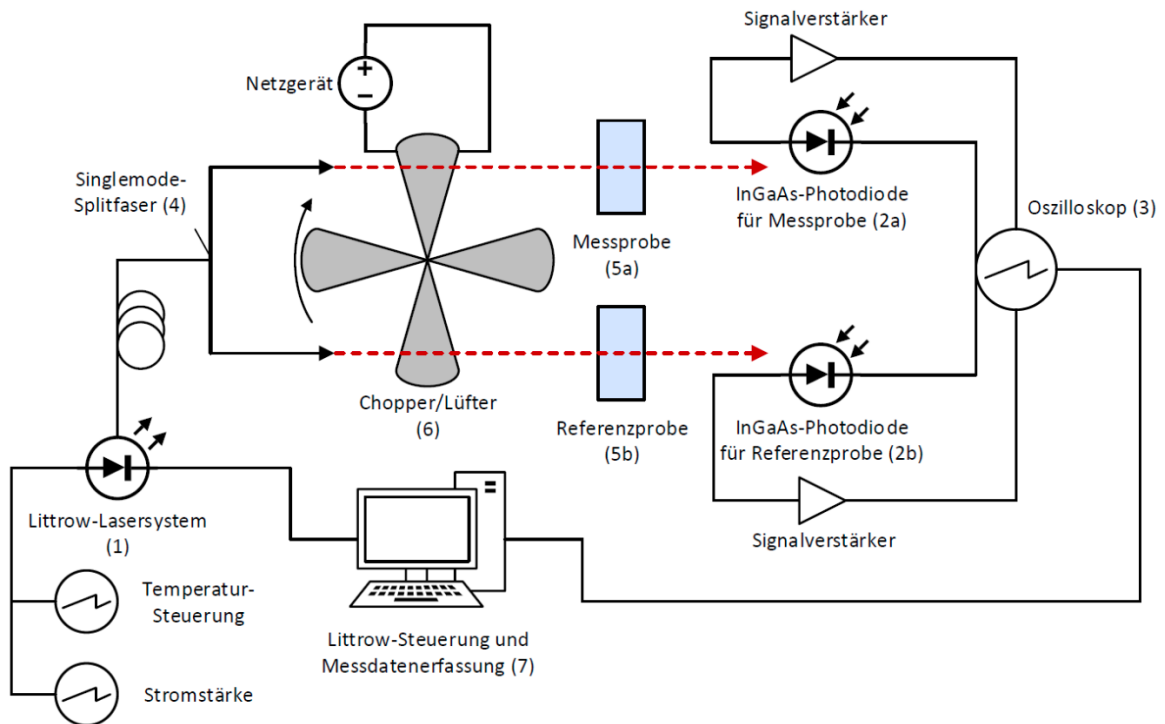


Figure 3.1: Experimental setup of the TDLAS measurement [1]

The procedure of the measurement is like following, whereas the laboratory script [1] is giving more detailed information.

1. Starting up sequence

For starting up the experiment, the laptop has to be boot up and the necessary software has to be started. Further, the control devices and operating temperatures have to be checked, such as laser temperature, laser current and voltage supply.

2. Calibration of the two measurement channels

Cuvettes have to be removed and a calibration measurement at each wavelength over both measurement paths A and B is needed. The calibration factor KF is calculated to normalize both signal pathways (especially the photodiodes). This step makes both pathways comparable.

3. Absorption spectrum measurement

As last step, the actual measurement of both species is carried out. Pathway A is used for acquiring the absorption spectrum of the species, path B is used with an empty cuvette as a reference measurement. Important to note is the dark signal of both channels.

3.2 Post-processing strategy

Starting off with the raw signals from the A/D-converter, the signal has to be corrected and normalized. Due to known and discussed effects of dark signals and different signal intensities from photo diodes, e.g. from differences out of manufacturing, two calibration steps have to be carried out. The dark signal must be subtracted from the general intensity (following Equation 3.1), the two channels have to be calibrated to each other with the help of an wavelength dependent calibration factor KF (see Equation 3.2).

$$I_{\text{measurement}} = I_{\text{photodiode}} + I_{\text{dark}} \quad (3.1)$$

$$KF(\lambda) = \frac{I(\lambda)}{I_0(\lambda)} \quad (3.2)$$

The calibration has to be considered for each discrete wavelength, the dark signal is uniformly relevant over the complete spectrum.

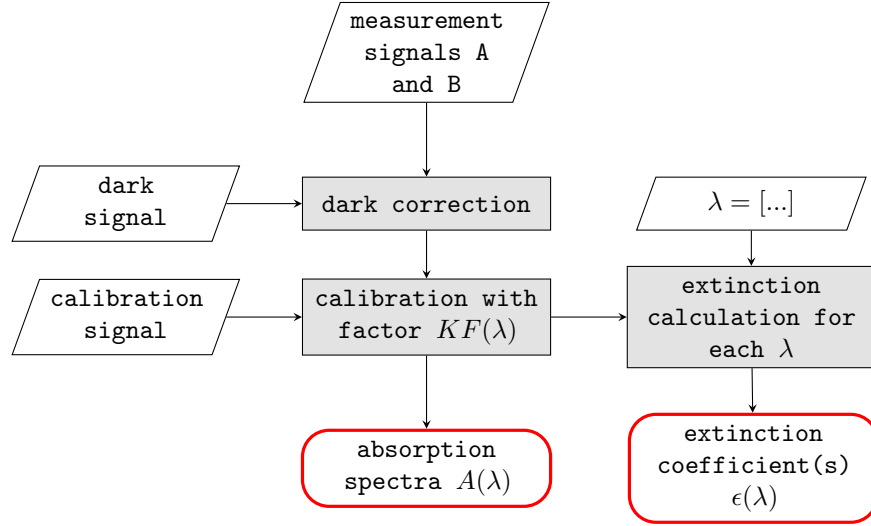


Figure 3.2: Evaluation procedure diagram

Since the Transmission ratio T , depending on the laser shot intensity I_0 and the transmitted intensity I is given through

$$T = \frac{I}{I_0}$$

and the Absorption ratio with the subtraction of T from one, we can state:

$$A = 1 - \frac{I}{I_0}$$

Further taking into account both calibration steps, we can develop as Absorption spectra

$$A(\lambda) = 1 - \frac{(I_A - I_{\text{dark}}) \cdot KF(\lambda)}{I_B - I_{\text{dark}}}. \quad (3.3)$$

Determining the extinction coefficient is possible through rearranging Lambert-Beer's law (see Equation 3.4). Important to note is the wavelength and temperature dependency of this coefficient, although the last one is kept constant at this experiment. Selected for evaluation of $\epsilon(\lambda)$ are following wavelengths:

$$\lambda = [1906, 1950, 1977, 2003] \text{ nm}$$

$$\epsilon(\lambda) = \frac{\log(I_0) - \log(I)}{c \cdot d} \quad (3.4)$$

The evaluation procedure is shown in Figure 3.2. For this calculation following parts have to be recorded during the experiment:

1. Measurement signal of the species at every wavelength
2. Reference spectrum at every wavelength
3. Dark signal before the measurement
4. Simultaneous signal of path A and B at every wavelength

4 Results

Plots:

- calibration curve over lambda
- absorption spectra
- laser signal, absorption signal, transmission signal over lambda
- extinction coefficient

4.1 Evaluation

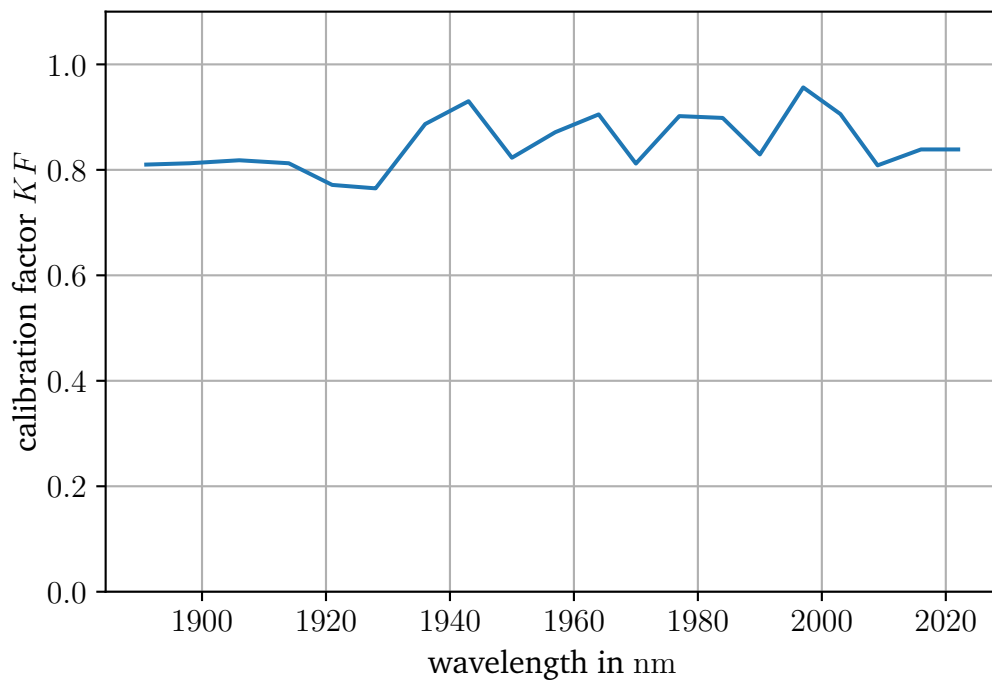


Figure 4.1: Calibration factor over wavelength

4.2 Error discussion

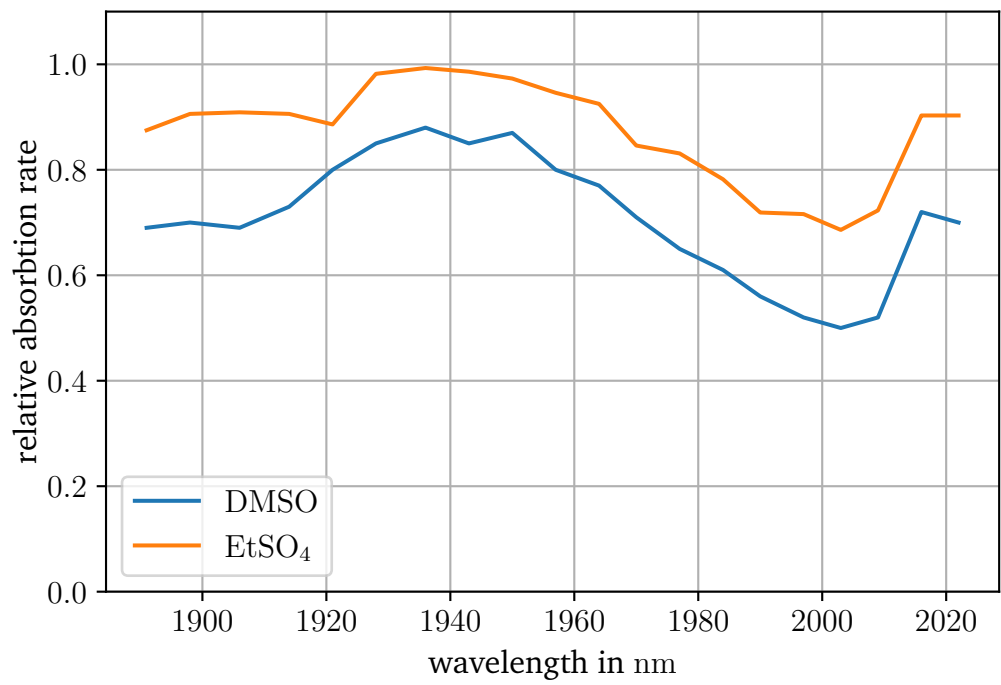


Figure 4.2: Relative absorption behavior of DMSO and EtSO₄ over the analyzed spectrum

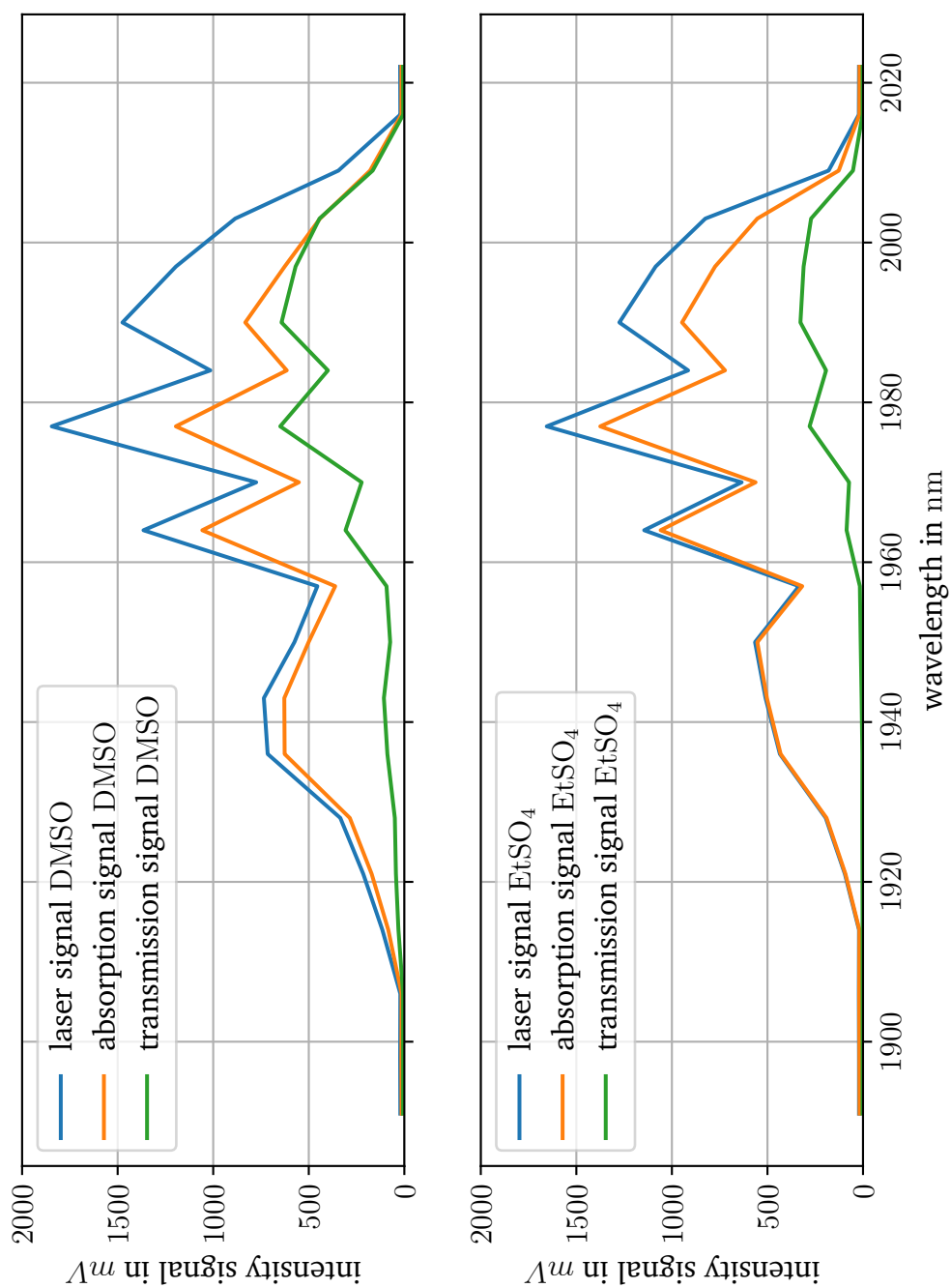


Figure 4.3: Comparison of laser signal intensity, transmission intensity, and absorption intensity; top for DMSO and bottom for EtSO₄ over the analyzed spectrum

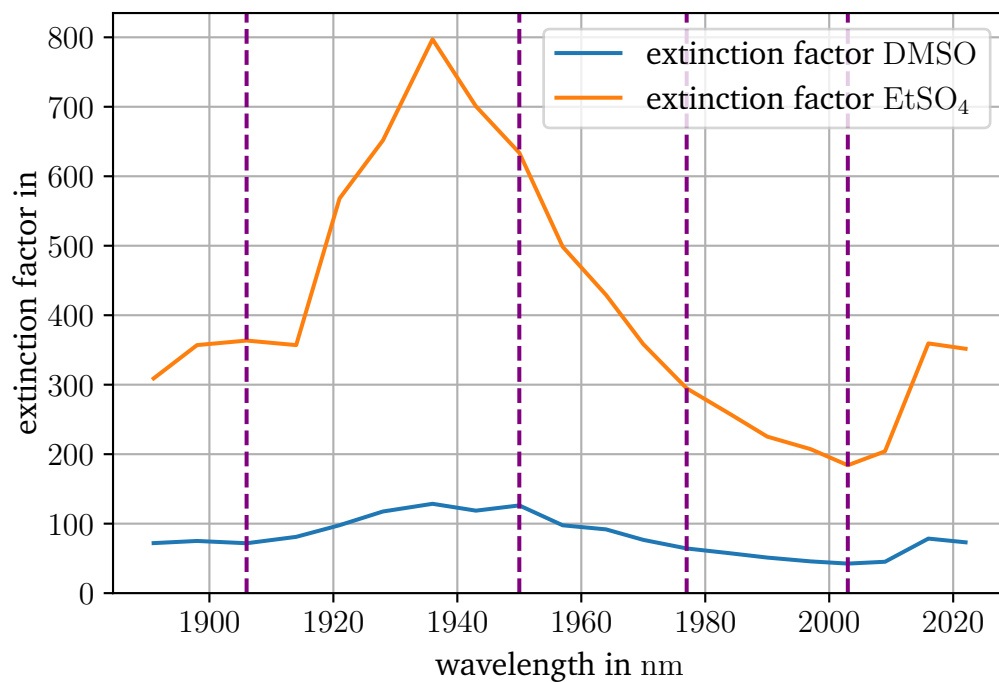


Figure 4.4: Extinction factor of DMSO and EtSO₄ over the analyzed spectrum

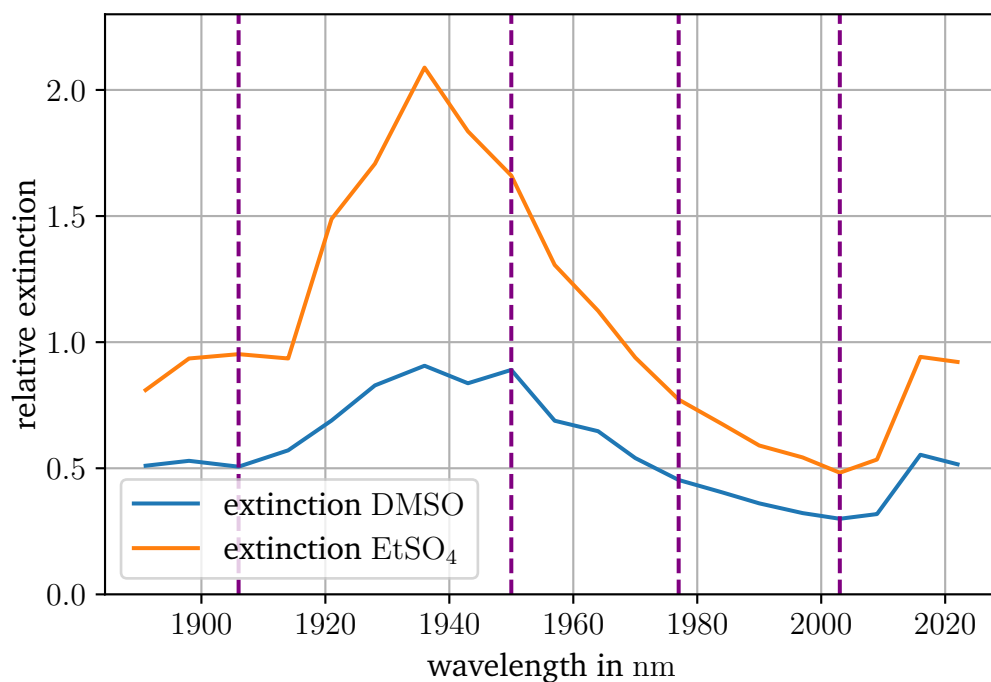


Figure 4.5: Extinction of DMSO and EtSO₄ over the analyzed spectrum

5 Summary

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